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APPLICATION NO.	FILING DATE	FIRST NAMED INVENTOR	ATTORNEY DOCKET NO.	CONFIRMATION NO.
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10/811,505

03/26/2004

Gerald D. Surender

03108/0201073-US0

8917

7278

7590

09/06/2007

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EXAMINER

HANOR, SERENA L

ART UNIT

PAPER NUMBER

1709

MAIL DATE

DELIVERY MODE

09/06/2007

PAPER

**Please find below and/or attached an Office communication concerning this application or proceeding.**

The time period for reply, if any, is set in the attached communication.

<b>Office Action Summary</b>	<b>Application No.</b> 10/811,505	<b>Applicant(s)</b> SURENDER ET AL.	
	<b>Examiner</b> Serena L. Hanor	<b>Art Unit</b> 1709	

-- The MAILING DATE of this communication appears on the cover sheet with the correspondence address --

**Period for Reply**

A SHORTENED STATUTORY PERIOD FOR REPLY IS SET TO EXPIRE 3 MONTH(S) OR THIRTY (30) DAYS, WHICHEVER IS LONGER, FROM THE MAILING DATE OF THIS COMMUNICATION.

- Extensions of time may be available under the provisions of 37 CFR 1.136(a). In no event, however, may a reply be timely filed after SIX (6) MONTHS from the mailing date of this communication.
- If NO period for reply is specified above, the maximum statutory period will apply and will expire SIX (6) MONTHS from the mailing date of this communication.
- Failure to reply within the set or extended period for reply will, by statute, cause the application to become ABANDONED (35 U.S.C. § 133). Any reply received by the Office later than three months after the mailing date of this communication, even if timely filed, may reduce any earned patent term adjustment. See 37 CFR 1.704(b).

**Status**

- 1) ☐ Responsive to communication(s) filed on \_\_\_\_.
- 2a) ☐ This action is **FINAL**.                      2b) ☒ This action is non-final.
- 3) ☐ Since this application is in condition for allowance except for formal matters, prosecution as to the merits is closed in accordance with the practice under *Ex parte Quayle*, 1935 C.D. 11, 453 O.G. 213.

**Disposition of Claims**

- 4) ☒ Claim(s) 1-21 is/are pending in the application.
- 4a) Of the above claim(s) \_\_\_\_ is/are withdrawn from consideration.
- 5) ☐ Claim(s) \_\_\_\_ is/are allowed.
- 6) ☒ Claim(s) 1-21 is/are rejected.
- 7) ☐ Claim(s) \_\_\_\_ is/are objected to.
- 8) ☐ Claim(s) \_\_\_\_ are subject to restriction and/or election requirement.

**Application Papers**

- 9) ☒ The specification is objected to by the Examiner.
- 10) ☐ The drawing(s) filed on \_\_\_\_ is/are: a) ☐ accepted or b) ☐ objected to by the Examiner.  
     Applicant may not request that any objection to the drawing(s) be held in abeyance. See 37 CFR 1.85(a).  
     Replacement drawing sheet(s) including the correction is required if the drawing(s) is objected to. See 37 CFR 1.121(d).
- 11) ☐ The oath or declaration is objected to by the Examiner. Note the attached Office Action or form PTO-152.

**Priority under 35 U.S.C. § 119**

- 12) ☐ Acknowledgment is made of a claim for foreign priority under 35 U.S.C. § 119(a)-(d) or (f).
- a) ☐ All    b) ☐ Some \* c) ☐ None of:
1. ☐ Certified copies of the priority documents have been received.
2. ☐ Certified copies of the priority documents have been received in Application No. \_\_\_\_.
3. ☐ Copies of the certified copies of the priority documents have been received in this National Stage application from the International Bureau (PCT Rule 17.2(a)).

\* See the attached detailed Office action for a list of the certified copies not received.

**Attachment(s)**

- |   |  |
|---|--|
| 1) <input checked="" type="checkbox"/> Notice of References Cited (PTO-892)   | 4) <input checked="" type="checkbox"/> Interview Summary (PTO-413)<br>Paper No(s)/Mail Date. <u>08/03/2007</u> . |
| 2) <input type="checkbox"/> Notice of Draftsperson's Patent Drawing Review (PTO-948)  | 5) <input type="checkbox"/> Notice of Informal Patent Application  |
| 3) <input checked="" type="checkbox"/> Information Disclosure Statement(s) (PTO/SB/08)<br>Paper No(s)/Mail Date <u>10/20/2004</u> . | 6) <input type="checkbox"/> Other: ____.   |

**DETAILED ACTION**

1. The claims 1-21 are pending and presented for examination.

***Specification***

2. The disclosure is objected to because of the following informalities:

general spelling, grammar, and usage mistakes throughout the entire specification.

p. 1 line 16: hitrogen

lines 28-29: is having

p. 2 line 1: some in commercial use and some in development

line 7: no comma needed

line 12: comma needed

line 22: comma needed

line 25: liquied, and

p. 3 lines 2-3: processes to synthesis of rutile grade titanium

line 4: show new liquid phase process,  $\text{TiO}_2$

line 5: 200-400 mm

line 10: precipitate

lines 12-13: However, when the solution is acidic, the hydrolysis product is a sol, in order to obtain rutile  $\text{TiO}_2$  by drying the gel at 40-50°C.

line 14: he

line 29: avaoids

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lines 31-33: corrosion of reactor material of construction and operational problems, mainly due to the high temperatures and corrosive gases involved.

p.4 line 2: phse

p. 5 line 34: In the drawings accompanying this specification

p. 6 line 1: rutiel

line 2: vapore

lines 4-5: of reactants of reactants

line 9: non-requirement of a need

lines 10-11: The present invention has successfully led high purity oxygen as in the chloride process.

line 12: development of to new titanium dioxide

line 14: particle characteristics such s particle shase

line 21: of three basis steps

line 28-29: it is envisioned that the present invention encompasses the full range

line 32: The reactor comprises of a

lines 33, 34: Inconel---this is a registered trademark

p. 7 line 1: the, comma needed

line2: respectively

lines 3, 5, 7, 19, 26: Inconel is a registered trademark

line 12:  $\text{TiCl}_4$

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- line 16: of from
- lines 23-24: includes a dopant material, in vapor phase, it positively affect  
the physical attributes of the titanium dioxide formed
- line 27: comma needed
- p. 8 line 4: amorphosúse, comma needed
- line 5: Teflon is a registered trademark
- line 11: there of
- line 13: temperature is reduced to as much as
- lines 18-19: calcinations temperature range between 500 to 700°C
- lines 23, 32:  $TiCl_4$
- line 24: urtile
- line 28: etrachloride
- p. 9 line 4: Teflon is a registered trademark
- lines 4-6: titanium dioxide powder/dry powders disagreement
- line 5: exhaust
- line 7: A Rotameter
- lines 9-10: fragment sentence
- lines 22, 24: powders
- p. 10 lines 9-10: no verb
- lines 13-14: too many verbs
- line 14: Table 4 show the specific areas of the powders
- p. 11 line 5: calcinations temperature

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line 8:       there of

line 10:       The other reactant involved in the process is water and  
                 ethanol

lines 16-17: lack of commas-comma splices

line 20:       1. The energy for cryogenic separation of air into high purity  
                 oxygen

line 23:       comma splice-pure oxygen, and the

Appropriate correction is required.

3.     The use of the trademark Inconel has been noted in this application. It should be capitalized wherever it appears and be accompanied by the generic terminology.

Although the use of trademarks is permissible in patent applications, the proprietary nature of the marks should be respected and every effort made to prevent their use in any manner which might adversely affect their validity as trademarks.

***Priority***

4.     Applicant is advised of possible benefits under 35 U.S.C. 119(a)-(d), wherein an application for patent filed in the United States may be entitled to the benefit of the filing date of a prior application filed in a foreign country. During the prior art search, the applicants' published work, "Low temperature process for the synthesis of rutile phase titania through vapor phase hydrolysis" in Journal of Materials Science vol. 40, 2005, 2999-3001, contained a reference to WO 2003-IN429, filed on 12/31/2003, published on 07/14/2005.

***Claim Rejections - 35 USC § 112 1<sup>st</sup>***

5. The following is a quotation of the first paragraph of 35 U.S.C. 112:

The specification shall contain a written description of the invention, and of the manner and process of making and using it, in such full, clear, concise, and exact terms as to enable any person skilled in the art to which it pertains, or with which it is most nearly connected, to make and use the same and shall set forth the best mode contemplated by the inventor of carrying out his invention.

3. Claims 10, 21 are rejected under 35 U.S.C. 112, first paragraph, as failing to comply with the written description requirement. The claim(s) contains subject matter which was not described in the specification in such a way as to reasonably convey to one skilled in the relevant art that the inventor(s), at the time the application was filed, had possession of the claimed invention. "The temperature at the exit of the aerosol reactor [is] maintained at less than 100°C for obtaining titanium dioxide particles having anatase phase" recited by claim 10, and "hydrolyzing  $\text{TiCl}_4$ ,  $\text{H}_2\text{O}$  and dopant in vapour phase mixture in a continuous aerosol reactor under non-isothermal conditions at temperature in the range 80 to 135°C" recited by claim 21 (b) are not consistent with examples 1 and 2 and Tables 3 and 5 in the specification. They disclose an exit gas stream temperature of 130-150°C and 137°C for obtaining anatase phase titanium dioxide particles, inconsistent with claim 10. Furthermore, Example 1 discloses a temperature range of 70-150°C, and Example 2 states obtaining rutile phase from amorphous phase at such temperatures only, inconsistent with claim 21.

***Claim Rejections - 35 USC § 103***

6. The following is a quotation of 35 U.S.C. 103(a) which forms the basis for all obviousness rejections set forth in this Office action:

(a) A patent may not be obtained though the invention is not identically disclosed or described as set forth in section 102 of this title, if the differences between the subject matter sought to be patented and

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the prior art are such that the subject matter as a whole would have been obvious at the time the invention was made to a person having ordinary skill in the art to which said subject matter pertains. Patentability shall not be negated by the manner in which the invention was made.

7. The factual inquiries set forth in *Graham v. John Deere Co.*, 383 U.S. 1, 148 USPQ 459 (1966), that are applied for establishing a background for determining obviousness under 35 U.S.C. 103(a) are summarized as follows:

1. Determining the scope and contents of the prior art.
2. Ascertaining the differences between the prior art and the claims at issue.
3. Resolving the level of ordinary skill in the pertinent art.
4. Considering objective evidence present in the application indicating obviousness or nonobviousness.

8. Claims 1, 3-6 are rejected under 35 U.S.C. 103(a) as being unpatentable over US 4,574,078 (US'078, hereafter) in view of Ahonen et al., and Rubio et al.

Claims 1, 3-6 are drawn to "a process for synthesis of ultrafine rutile phase titanium dioxide particles through vapor phase hydrolysis of titanium tetrachloride comprising the step of (a) hydrolyzing a mixture of  $\text{TiCl}_4$  and  $\text{H}_2\text{O}$  and a dopant in vapour phase in an aerosol reactor; (b) collecting amorphous or anatase titanium dioxide powder formed as dry powders; (c) calcining the dry powder to obtain rutile phase titanium dioxide." Said dopant is ethanol. The molar ratio of water to titanium tetrachloride in the feed is in the range 10 to 15, the molar concentration of the dopant is 1-10 based on water vapor, and the reaction mixture contains from 1-10% mole ethanol to  $\text{TiCl}_4$ .

US'078 is drawn to a process for preparing particles of metal oxides by reacting a hydrolyzable metal with steam, used in an amount corresponding to about 1.5 to 12 times the stoichiometric ration with respect to the hydrolyzable metal compound, and an



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inert gas to form an aerosol of liquid particles which then become solid particles, which are then separated from the gas and calcined to obtain the corresponding metal oxide.

US'078 differs from the claim in that it fails to teach the use of a dopant in order to achieve substantial reduction of the calcinations temperature required for obtaining titanium dioxide particles with rutile phase.

It would have been obvious to one of ordinary skill in the art at the time the invention was made to modify the process of US'078 by using a dopant as taught by Ahonen et al. and Rubio et al. Ahonen et al. is drawn to a droplet-to-particle aerosol synthesis that uses a quartz aerosol reactor with two concentric tubes. Rubio et al. is drawn to the vapor phase hydrolysis of titanium tetrabutoxide (TTB), water, and nitrogen, with and without alcohol, with a reactor wall temperature of 200°C. Furthermore, the claimed molar concentration of dopant to water vapor and the reaction mixture can be arrived at by routine optimization.

Put another way, Rubio et al. teaches the molar concentration of dopant to water vapor to be an art recognized result-effective variable, depending on the type of material to be used. It would have been obvious to one having ordinary skill in the art at the time the invention was made to modify said concentration ratio. Alternatively, it would have been obvious to one of ordinary skill in the art at the time of the invention to choose the instantly claimed ranges through process optimization, since it has been held that if the general conditions of a claim are disclosed in the prior art, discovering the optimum or workable ranges involves only routine skill in the art. See *In re Boesch*, 205 USPQ 215 (CCPA 1980).

One would have been motivated to make such a modification of adding a dopant in order to reduce the calcinations temperature for economic reasons for industrial applicability as well as to reduce the particle size to obtain rutile phase titanium dioxide. A dopant has been used previously to obtain such results, i.e. smaller particle size and reduced calcinations temperature, and the use of different sources of titanium and/or different aerosol processes is obvious to one of ordinary skill. A range can be disclosed in multiple prior art references instead of in a single prior art reference depending on the specific facts of the case. See *Iron Grip Barbell Co., Inc. v. USA Sports, Inc.*, 392 F.3d 1317, 1322, 73 USPQ2d 1225, 1228 (Fed. Cir. 2004). Therefore, said claims are obvious and not patentably distinct over the prior art of the record.

9. Claims 1, 7-9, 11, 15-18 are rejected under 35 U.S.C. 103(a) as being unpatentable over US 5,698,177 (US'177, hereafter) in view of Richard et al. and Xia et al.

Claims 1, 7-9, 11, 15-18 are drawn to a process for synthesis of ultrafine rutile phase titanium dioxide particles through vapor phase hydrolysis of titanium tetrachloride, wherein the flow rate of  $\text{TiCl}_4$  is in the range of 10-200  $\text{cm}^3/\text{min}$ , the  $\text{TiCl}_4$  vapor concentration inside the reactor is  $7 \times 10^{-4} - 1 \times 10^{-2}$  mol/min, the flow rate of water vapor is 240-1500  $\text{cm}^3/\text{min}$ , the aerosol reactor is externally heated in order to avoid particle coating on the walls, the dopant is an inert gas, vapor phase  $\text{TiCl}_4$  is formed by bubbling an inert gas through  $\text{TiCl}_4$  liquid, the molar ratio of water to titanium tetra chloride in the feed is in the range 10-15, and the water vapor is formed by bubbling air or inert gases through water.

US'177 teaches a process that uses an aerosol flame reactor where  $\text{TiCl}_4$  and  $\text{O}_2$  are mixed in the vapor phase, heated by a flame formed by combustion of a hydrocarbon fuel, and the powder is ultimately collected in a corona electric field formed between two needle electrodes. An inert gas such as nitrogen is bubbled through  $\text{TiCl}_4$  liquid to form vapor phase  $\text{TiCl}_4$ , which is then fed into the center core of the reactor at a flow rate of  $100\text{-}300\text{ cm}^3/\text{min}$  and a concentration of  $7 \times 10^{-5} - 1 \times 10^{-2}\text{ mol/min}$ . The  $\text{O}_2$  is further fed into a sleeve of said reactor adjacent to the center core, and the fuel is fed into the reactor in a sleeve which surrounds both sleeves.

US'177 differs from the instant claims in that it fails to teach the use of a dopant, water vapor, an externally heated Inconel™ concentric reactor, and calcination of the collected powder.

It is obvious to one of ordinary skill in the art to vaporize said dopant by bubbling said inert gas through it just as is done with the  $\text{TiCl}_4$ . In addition, bubbling the inert gas to form water vapor is an obvious step from bubbling it through the  $\text{TiCl}_4$ . Furthermore, externally heating the reactor to avoid thermophoresis inside the reactor but not limit such upon exiting the reactor is also known.

Xia et al. teaches a reactor made of two concentric glass tubes that is externally heated in a vertical furnace, wherein the  $\text{H}_2\text{O}:\text{TiCl}_4$  ratio is 10:1. Furthermore, no dopant is used, and the product is collected by thermophoresis and glass-fiber filter at the reactor exit. Finally, Richard et al. recites the use of an ultrasonic generator used for droplet atomization in the in-droplet hydrolysis, where TTNB is used, and the carrier gas

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(nitrogen and pressurized air) was bubbled through ethanol prior to droplet dispersion in order to avoid solvent evaporation in the single-tube aerosol generator.

The motivation to combine above said references is within the scope of a person of ordinary skill in the art because adding a dopant helps to reduce particle size and calcination temperature. Also, if three substances are being mixed, e.g.  $\text{TiCl}_4$ ,  $\text{H}_2\text{O}$ , and ethanol, it is obvious to use a reactor with three concentric tubes instead of only two. It is further obvious to use the inert gas to vaporize the  $\text{TiCl}_4$ ,  $\text{H}_2\text{O}$ , and ethanol. Therefore said claims are obvious and not patentably distinct over the prior art of the record.

10. Claims 1, 12-14, 19 are rejected under 35 U.S.C. 103(a) as being unpatentable over US 1,931,380 (US'380, hereafter) in view of Wegner et al and Richard et al.

Claims 1, 12-14, 19 are drawn to a process for synthesis of ultrafine rutile phase titanium dioxide particles through vapor phase hydrolysis of titanium tetrachloride, wherein the aerosol reactor comprises a 3-tube concentric jet assembly wherein  $\text{TiCl}_4$  is introduced into the innermost tube, dopant is introduced into the outermost tube, and water vapor is introduced into the middle tube, the concentric tubes are made of Inconel™ at the entrance of the reactor, and the reactor wall temperature is from 200-450°C.

US'380 teaches the production of titanium dioxide from vaporized titanium tetrachloride in the presence of steam or water vapor. Air is bubbled through  $\text{TiCl}_4$  and water separately, and the two gaseous streams enter a vertical reaction vessel made of a material capable of resisting hydrochloric acid and externally heated to 300-500°C.

US'380 differs from the instant claims in that it fails to teach the use of a dopant or an inert gas.

It is obvious to one of ordinary skill in the art to add a third concentric tube to the reactor if using a dopant when US'380 is considered in view of Wegner and Richard et al. Wegner et al. recites a process for the synthesis of titania nanoparticles via a flame aerosol reactor. The reactor consists of three concentric stainless-steel tubes. An argon stream carrying titanium-tetra-isopropoxide (TTIP) is introduced through the center tube, methane flows through the first annulus, and oxygen through the outer annulus. Finally, Richard et al. recites the use of an ultrasonic generator used for droplet atomization in the in-droplet hydrolysis, where TTNB is used, and the carrier gas (nitrogen and pressurized air) was bubbled through ethanol prior to droplet dispersion in order to avoid solvent evaporation in the single-tube aerosol generator. The wall temperature of said generator is 350-600°C. Furthermore, stainless steel is an iron-carbon alloy that does not easily stain, corrode or rust, so the use of Inconel™, a nickel-based superalloy that is oxidation and corrosion resistant and typically used in high temperature applications, is considered based on routine optimization. See *Ex parte Wu*, 10 USPQ 2031 (Bd. Pat. App. & Inter. 1989). The reactor walls must be resistant to the effects of hydrochloric acid. Finally, a wall temperature chosen from the range of 200-450°C is also drawn from 300-500°C and 350-600°C based on routine optimization in order to avoid particle coating on the walls through thermophoresis. A range can be disclosed in multiple prior art references instead of in a single prior art reference

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depending on the specific facts of the case. *Iron Grip Barbell Co., Inc. v. USA Sports, Inc.*, 392 F.3d 1317, 1322, 73 USPQ2d 1225, 1228 (Fed. Cir. 2004).

Put another way, Richard et al. teaches the temperature range of the external heating applied to the reactor to be an art recognized result-effective variable, depending on the type of material to be used. It would have been obvious to one having ordinary skill in the art at the time the invention was made to modify the temperature. Alternatively, it would have been obvious to one of ordinary skill in the art at the time of the invention to choose the instantly claimed ranges through process optimization, since it has been held that if the general conditions of a claim are disclosed in the prior art, discovering the optimum or workable ranges involves only routine skill in the art. See *In re Boesch*, 205 USPQ 215 (CCPA 1980).

The motivation to combine above said references is within the scope of a person of ordinary skill in the art because adding a dopant helps to reduce particle size and calcination temperature. Also, if three substances are being mixed, e.g.  $\text{TiCl}_4$ ,  $\text{H}_2\text{O}$ , and ethanol, it is obvious to use a reactor with three concentric tubes instead of only two. Therefore said claims are obvious and not patentably distinct over the prior art of the record.

11. Claims 1-2, 10, 21 are rejected under 35 U.S.C. 103(a) as being unpatentable over US 4,241,042 (US'042, hereafter) in view of US 5,846,511 (US'511, hereafter) and US 1,931,380 (US'380, hereafter).

Claims 1-2, 10, 21 are drawn to a process for synthesis of ultrafine rutile phase titanium dioxide particles through vapor phase hydrolysis of titanium tetrachloride,

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wherein the amorphous particles are calcined at a temperature range of 150-400°C, anatase particles are produced when the exit temperature of the aerosol reactor is maintained at less than 100°C, and the temperature range of the aerosol reactor is further maintained at 80-135°C.

US'042 teaches a method for preparing titanium dioxide via a liquid aerosol of water vapor and titanium tetrachloride that is heated before and after recovery from the reactor at a temperature of 150-1,100°C, and said method produces particles with a diameter of 50-3,000 nm.

US'042 differs from the instant claims in that it does not use a dopant, and the product is heated before and after recovery at a temperature range that extends beyond that of the instant claims. In addition, it does not specify the crystalline shape, only the particle size.

It is obvious to one of ordinary skill to combine the processes, since US'042 specifies the crystal type and the calcination temperature range 150-1,100°C and US'511 specifies the particle size, the use of a dopant, and the calcination temperature ranges 150-200°C and 600-800°C. US'380 specifies the calcination temperature range 200-400°C for a general method to produce titanium dioxide powder. US'511 recites a process for preparing anatase-phase titania powders where titanium tetrachloride, water, and a lower alcohol are heated to form amorphous precipitates of titanium hydroxide, which is then calcined to produce the anatase phase. US'380 teaches the production of titanium dioxide from vaporized titanium tetrachloride in the presence of steam or water vapor. The particles pass from the reactor to a dust chamber, which is

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heated to 200-400°C to inhibit condensation or adsorption of hydrochloric acid on or by the titanium dioxide as same is precipitating. Generally, differences in concentration or temperature will not support the patentability of subject matter encompassed by the prior art unless there is evidence indicating such concentration or temperature is critical.

"[W]here the general conditions of a claim are disclosed in the prior art, it is not inventive to discover the optimum or workable ranges by routine experimentation." *In re Aller*, 220 F.2d 454, 456, 105 USPQ 233, 235 (CCPA 1955).

Put another way, US'042 and US'511 teach the calcination temperature to be an art recognized result-effective variable, depending on the type of material to be used. It would have been obvious to one having ordinary skill in the art at the time the invention was made to modify said temperature to obtain the desired results. Alternatively, it would have been obvious to one of ordinary skill in the art at the time of the invention to choose the instantly claimed ranges through process optimization, since it has been held that if the general conditions of a claim are disclosed in the prior art, discovering the optimum or workable ranges involves only routine skill in the art. See *In re Boesch*, 205 USPQ 215 (CCPA 1980).

The motivation to combine above said references is within the scope of a person of ordinary skill in the art because adding a dopant helps to reduce particle size and calcination temperature. Additionally, using a slightly higher hydrolysis temperature than that employed by US'511 may help to lower the calcination temperature used to produce the rutile powder, based on routine optimization. Therefore said claims are obvious and not patentably distinct over the prior art of the record.



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**Conclusion**

12. No claim is allowed.

4. Any inquiry concerning this communication or earlier communications from the examiner should be directed to Serena L. Hanor whose telephone number is (571) 270-3593. The examiner can normally be reached on Monday - Thursday 8:00 AM - 5:30 PM EST. The examiner can also be reached on alternate Fridays.

If attempts to reach the examiner by telephone are unsuccessful, the examiner's supervisor, Vickie Kim can be reached on (571) 272-0579. The fax phone number for the organization where this application or proceeding is assigned is 571-273-8300.

Information regarding the status of an application may be obtained from the Patent Application Information Retrieval (PAIR) system. Status information for published applications may be obtained from either Private PAIR or Public PAIR. Status information for unpublished applications is available through Private PAIR only. For more information about the PAIR system, see <http://pair-direct.uspto.gov>. Should you have questions on access to the Private PAIR system, contact the Electronic Business Center (EBC) at 866-217-9197 (toll-free). If you would like assistance from a USPTO Customer Service Representative or access to the automated information system, call 800-786-9199 (IN USA OR CANADA) or 571-272-1000.

SLH

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